IS 6940 (1982): Methods of Test for Pesticides and their Formulations [FAD 1: Pesticides and Pesticides Residue Analysis]

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IS : 6940 - 1982
(Reaffirmed - 2012)

Indian Standard

METHODS OF TEST FOR PESTICIDES AND THEIR FORMULATIONS

(First Revision)

Fourth Reprint NOVEMBER 2002

UDC 632.951:620.1

BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

Gr 8 *September* 1982

Indian Standard

METHODS OF TEST FOR PESTICIDES AND THEIR FORMULATIONS

(First Revision)

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AMENDMENT NO. 1 SEPTEMBER 1988
TO
IS : 6940 - 1982 METHODS OF TEST FOR PESTICIDES AND THEIR FORMULATIONS

(First Revision)

(*Page* 20, *clause* **11.4.2**) — Substitute the following for the existing clause :

'**11.4.2** *Procedure* — Weigh accurately 2 g of the material and pour uniformly and quickly in the beaker containing 100 ml of the standard hard water (*see* **11.2.2.1**). Start the stop-watch simultaneously. Note the time when the bulk material is completely submerged into water.'

(*Page* 26, *clause* **13.5.4**) — Substitute the following for the existing clause :

'**13.5.4** *Electrometric Procedure for Determination of Acidity or Alkalinity.*

13.5.4.1 *Reagents*

a) *Methyl alcohol* — distilled.

b) *Sodium hydroxide* — 0·05 N.

c) *Hydrochloric acid* — 0·05 N.

13.5.4.2 *Apparatus*

a) *p*H meter.

13.5.4.3 *Procedure* — Weigh accurately about 2 g of the sample in a conical flask. Add 25 ml methyl alcohol and stir for 5 minutes. Filter through a Buchner funnel into the filter flask. Rinse the conical flask and the Buchner funnel with methyl alcohol (3×5 ml). Transfer the combined methyl alcohol extracts to the titration vessel rinsing the filter flask with 10 ml of methyl alcohol. Add 10 ml of distilled water and titrate electrometrically with sodium hydroxide solution or hydrochloric acid to *p*H 5 at 27°C. Calculate the acidity or alkalinity whatsoever by the factor given in **13.5.2.3** or **13.5.3.3**.

(AFCDC 6)

Printed at New India Printing Press, Khurja, India

AMENDMENT NO. 2 OCTOBER 2003
TO
IS 6940 : 1982 METHODS OF TEST FOR PESTICIDES AND THEIR FORMULATIONS

(First Revision)

(*Page* 22, *clause* **13.3.2.3**, *after line* 11) — Insert 'close the cylinder with the stopper and invert it sharply to give 10 complete cycles within 20 seconds'.

(FAD 1)

Reprography Unit, BIS, New Delhi, India

Indian Standard

METHODS OF TEST FOR PESTICIDES AND THEIR FORMULATIONS

(First Revision)

0. FOREWORD

0.1 This Indian Standard was adopted by the Indian Standards Institution on 26 February 1982, after the draft finalized by the Pest Control Sectional Committee had been approved by the Agricultural and Food Products Division Council and the Chemical Division Council.

0.2 This standard was originally published in 1973 and an amendment was issued subsequently. Since then a number of Indian Standards on pesticides have been published and references made in those standards to the methods of test prescribed in this standard. In the light of experience gained during the last few years, it became necessary to issue revised version of this standard. This revised version covers comprehensively the general analytical details and methods which were not covered in the original standard. Besides, general methods of test for granules have also been included.

0.3 About 200 Indian Standard specifications for technical grade pesticides and their formulations have been published. The standards for technical grade pesticides include insecticides, fungicides, herbicides, rodenticides, and fumigants. The Indian Standard specifications for different kinds of formulations for the above pesticides include emulsifiable concentrates, water dispersible powders, dusting powders, granules, seed dressing formulations and concentrates such as bait concentrates and ready-to-use concentrates of certain pesticides themselves. All these standards for the technical grade as well as for the formulations include certain methods of test that are common to all these standards. With a view to avoid the repetition and to provide an easy reference, it was found desirable to publish this standard as applicable generally to various technical grade pesticides and their formulations.

0.3.1 While preparing this standard, due consideration has been given to the methods of test as arrived at in the WHO specification; and also the methods of test, prepared by the Collaborative Analytical Pesticides Committee of the Food and Agricultural Organization.

0.4 In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS : 2-1960*.

1. SCOPE

1.1 This standard specifies general methods of test for technical grade pesticides and their different formulations.

2. QUALITY OF REAGENTS

2.1 Unless specified otherwise, pure chemicals and distilled water (*see* IS: 1070-1977†) shall be employed in tests.

NOTE — ' Pure chemicals ' shall mean chemicals that do not contain impurities which affect the results of analysis.

SECTION 1 GENERAL METHODS OF TEST FOR PESTICIDES, TECHNICAL GRADE

3. GENERAL

3.1 There are several methods which are generally applicable to all technical grade pesticides and they are given under various clauses.

4. DETERMINATION OF MOISTURE CONTENT

4.0 General — For the determination of moisture content, two methods, namely, the Karl Fisher method (*see* **4.1**) and the Dean and Stark method (*see* **4.2**) have been specified. The Karl Fisher method may be followed if the expected moisture content is below 1·0 percent and the Dean and Stark method in cases where the expected moisture content is 1·0 percent and above.

4.1 Karl Fischer Method — The procedure for determining moisture content by this method shall be in accordance with IS : 2362-1973‡.

NOTE — All necessary precautions shall be taken in order to avoid any possible hazards due to oxidation, while determining moisture content in mercaptan based pesticides.

4.2 Dean and Stark Method

4.2.1 *Apparatus*

4.2.1.1 *Distillation flask* — made of hard glass with 500 ml capacity and provided with a ground-glass socket.

*Rules for rounding off numerical values (*revised*).

†Specification for water for general laboratory use (*second revision*).

‡Determination of moisture content by the Karl Fischer method (*first revision*).

4.2.1.2 *Reflux condenser* — made of glass, water cooled, having the outside diameter of the inner tube 16 to 17 mm, and that of the jacket 23 to 25 mm. The lower end of the condenser is provided with a ground-glass cone. The tip of the condenser is ground at an angle of approximately 30° from the vertical axis.

4.2.1.3 *Receiver* (*or trap*) — made of hard glass and consisting essentially of an upper chamber, provided with a side-arm leading to the distillation flask, and a cylindrical graduated portion, the lower end of which is sealed. The opening of the upper chamber is provided with a ground-glass socket fitting the cone of the condenser. The lower end of the side-arm is provided with a ground-glass cone fitting the socket of the distillation flask. The graduated portion has a capacity of 5 ml when filled to the highest graduation mark. The scale covers the range 0 to 5 ml, with graduation marks at 0·1 ml, intervals. The error at any indicated capacity should not exceed 0·02 ml.

4.2.1.4 *Heat source* — either an oil-bath or an electric heater provided with a sliding rheostat or other means of heat control. The temperature of the oil in the bath should not be very much higher than the boiling point of petroleum naptha (*see* **4.2.2.2**).

4.2.1.5 *Copper wire* — long enough to extend through the condenser with one end twisted into a spiral. The diameter of the spiral should be such that it fits snugly within the graduated portion of receiver and yet can be moved up and down.

4.2.2 *Reagents*

4.2.2.1 *Chromic acid cleaning solution*

4.2.2.2 One of the following solvents may be used:

a) *Petroleum naphtha* — with a boiling range of 90 to 210°C;

b) *n-Heptane*, — boiling point around 98°C; and

c) *Toluene* — boiling point around 110°C.

4.2.3 *Procedure* — Clean the entire apparatus (*see* Fig. 1) with chromic acid solution to minimize the adherence of water droplets to the sides of the condenser and the receiver. Rinse thoroughly with distilled water and dry completely before using.

Place an appropriate amount of the sample accurately weighed into the distillation flask. The amount of sample taken should be such that the distillation flask is not more than one-third full; it should not contain more than 1·8 ml of water. Add sufficient volume of the solvent (100 to 200 ml) so that the sample shall be completely covered by the solvent while the distillation is in progress. Swirl to mix and add pumice stone

to ensure steady boiling. Assemble the apparatus and fill the receiver with the solvent by pouring the solvent through the condenser until it begins to overflow into the distillation flask. Attach a guard tube containing anhydrous calcium chloride at the top of the condenser to prevent condensation of atmospheric moisture within the tube. In order that the refluxing may be under control, wrap the distillation flask and the tube leading to the receiver with asbestos fibre. Heat the flask at such a rate that about 100 drops are distilled over, increase the distillation rate to about 200 drops/min and continue heating until no more water is collected. Purge the condenser occasionally during the distillation with 5-ml portions of the solvent in order to wash down any moisture adhering to the walls of the condenser. The water in the trap may be made to separate from the solvent by means of a copper wire, one end of which is twisted into a helix. The copper wire if moved up and down in the condenser and receiver occasionally causes the water to settle down at the bottom of the trap. Reflux until the water level in the receiver remains unchanged for 30 minutes and then shut off the source of heat. Flush the condenser with the solvent, making use of the copper wire to disengage any moisture droplets. Allow to stand for about 15 minutes and then read the volume of water.

4.2.4 *Calculation*

$$\text{Moisture content, percent by mass} = \frac{100\,V}{M}$$

where

V = volume in ml of water collected; and

M = mass in g of the material taken for the test.

4.2.5 *Precision* — The results of a duplicate determination of moisture content of the material, percent by mass, should not vary by more than $\pm$ 0·05 from that of the first determination.

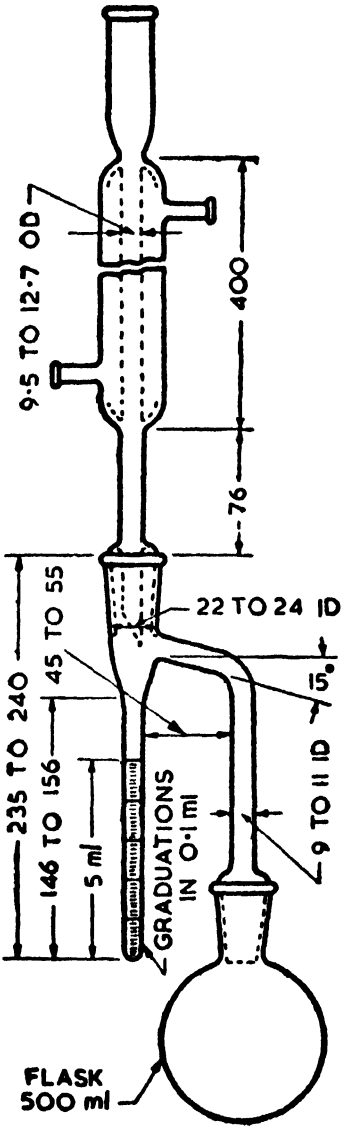
5. DETERMINATION OF RELATIVE DENSITY OF LIQUID PESTICIDES

5.1 Terminology — For the purpose of pesticides, the relative density of the material shall be the ratio of the mass in air of a given volume of the material generally at 27°C to that of an equal volume of water at the same temperature.

5.2 Apparatus

5.2.1 *Relative Density Bottle* — Castell Evans modification with ground-in stopper and ground-on cap (*see* Fig. 2) or a Perkin type pyknometer tube (*see* Fig. 3 and also IS : 5717-1970*).

*Specification for pyknometers.



All dimensions in millimetres.

FIG. 1 MOISTURE DISTILLATION APPARATUS

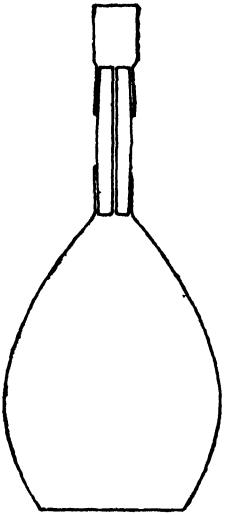


FIG. 2 SPECIFIC GRAVITY BOTTLE (CASTELL EVANS MODIFICATION WITH GROUND-IN STOPPER AND GROUND-ON CAP)

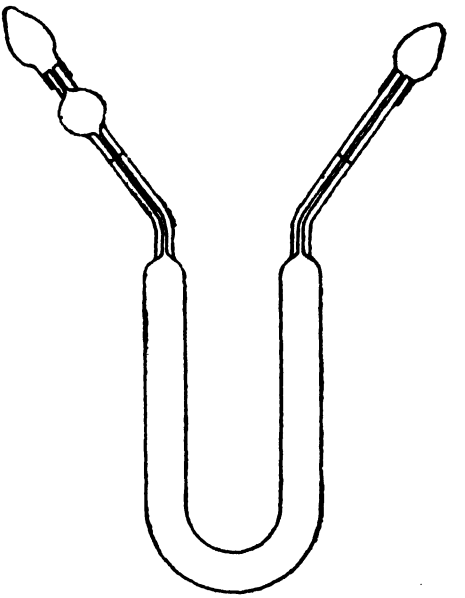


FIG. 3 PYKNOMETER TUBE (PERKIN'S TYPE)

5.3 Procedure — Weigh a clean and dry relative density bottle or a pyknometer of 25 ml capacity. Fill the tared relative density bottle or the pyknometer with freshly boiled water, place it in a bath maintained at 27 ± 1°C and allow sufficient time to attain the temperature of the bath. Then insert the ground-in stopper which has also been brought to 27 ± 1°C. Wipe off the excess from the stopper and place the cap in position. Remove the relative density bottle or the pyknometer from the bath, bring to room temperature and weigh. Empty the relative density bottle or the pyknometer, clean and dry it, and then repeat the operation with the material at 27 ± 1°C.

5.4 Calculation and Report

5.4.1 Specify the temperature of testing in the report.

5.4.2 The relative density is calculated by the following formula:

$$\text{Relative density at } 27°C/27°C = \frac{C - A}{B - A}$$

where

C = mass in g of the relative density bottle or the pyknometer filled with the material at 27°C,

A = mass in g of the dry relative density bottle or the pyknometer, and

B = mass in g of the relative density bottle or the pyknometer filled with water at 27°C.

6. DETERMINATION OF MELTING POINT

6.1 Apparatus

6.1.1 *Thermometer* — A long-stem and short-bulb thermometer with a range of 0 to 200°C or 100 to 200°C and with subdivisions of 0·5°C.

6.1.2 *Bath and Heating Assembly* — This consists of a suitable chamber with a glass front. Inside, there is a long-necked flask (250-ml Kjeldahl flask) three-fourths full with liquid paraffin and placed on a suitable mounting for heating with a gas burner. The flask is loosely fitted with a stopper which carries a thermometer. The chamber is provided with appropriate illumination to observe the melting of the material and the reading of temperature.

6.2 Procedure — Take a small quantity of finely powdered substance and dry the same in a vacuum desiccator over silica gel or phosphorus pentoxide for 24 hours or according to the requirements given in the

specification. Transfer the same in the capillary tube with one end sealed. Ensure proper packing of the material in the tube by gently tapping it on the table. The material in the capillary tube should be sufficient to cover the entire length of the thermometer bulb. Introduce the filled capillary tube into the heated bath at a temperature 10°C below the expected lower limit of the melting range. Attach the capillary tube with the thermometer such that the material is very close to the bulb. Introduce the thermometer in the bath in such a way that the thermometer bulb is well below the surface of the liquid. Now heat slowly and carefully so that the rise in bath temperature is about 1°C per minute. Note the temperature of initial melting (when first liquid drop appears) and completion of melting (when no solid particle is left). Report the temperature of melting point completion and the difference of temperatures as melting range.

NOTE — Ensure that two consecutive determinations do not differ by more than 0·5°C.

7. DETERMINATION OF SOFTENING POINT

7.1 Outline of Method — The softening point of technical grade pesticide is determined at a temperature when the test sample becomes sufficiently fluid to drop from the thermometer used in the determination of the softening point.

7.2 Apparatus

7.2.1 *Heating Vessel* — a glass beaker, 1 000 ml capacity.

7.2.2 *Stirrer* — capable of mixing the liquid thoroughly.

7.2.3 *Thermometer* — 0 to 200°C with 1°C subdivisions.

7.2.4 *Sample-Melting Bath* — an oil-bath maintained at 125 ± 1°C.

7.2.5 *Test-Tubes* — provided with rims; 175 mm long and 22 mm outside diameter.

7.3 Procedure

7.3.1 Place sufficient material which, when melted, will fill in one-third or one-half of 50-ml beaker. Place the beaker in a sample-melting bath for 4 to 8 minutes until the material is melted completely. The melted sample shall be clear and free of crystals.

7.3.2 Weigh the thermometer to the nearest 0·01 g and warm over a flame or hot-plate until it is heated to about 50°C. Immediately dip the thermometer vertically into the melted sample until the shoulder of the bulb is covered. Lift the thermometer from the sample, hold it in a

vertical position keeping the bulb on the top, rotate until the sample solidifies and weigh. The mass of the sample on the thermometer bulb should be between 0·50 and 0·55 g. If there is insufficient material on the thermometer, dip it again into the melted sample to pick up the desired quantity of the material. If the bulb of the thermometer has picked up too much sample repeat the operation. Allow the thermometer and the sample to cool until they reach approximately room temperature which takes about 15 minutes.

7.3.3 Adjust the heating vessel containing liquid paraffin to 110 to 111°C. Insert the thermometer carrying the sample in the test-tube supporting it with a notched rubber or cork stopper so that the lower end of the bulb is about 25 mm from the bottom of the tube. Place the tube in the vessel so that the bottom of the tube is about 25 mm above the bottom of the vessel. The bottom of the thermometer bulb is thus 50 mm above the bottom of the beaker and the top of the bulb about 25 mm below the level of the liquid in the vessel. Stir the vessel continuously during the test with a motor stirrer.

7.3.3.1 Observe the thermometer carrying the sample inside the test-tube and record the temperature of the softening point from the thermometer, when the first drop of the melted material falls from the end of the bulb.

7.3.4 Repeat the operation specified in **7.3.3** and **7.3.3.1** until the results agree within 1°C (*see* Note).

NOTE — Two or more samples may be placed in the vessel during the same operation.

8. DETERMINATION OF SETTING POINT

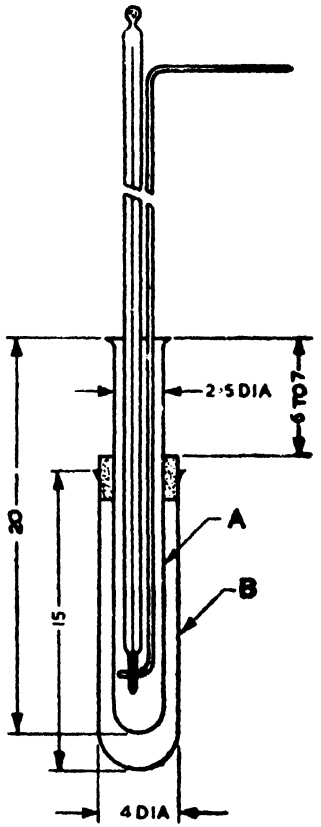
8.1 Apparatus (*see* Fig. 4)

8.1.1 *Boiling Tube A* — a 20 ± 0·1 mm thick-walled, 20 × 2·5 cm tube fitted with a cork collar at 6 to 7 cm below the top edge of the tube, the collar fitting tightly in the boiling tube *B* (*see* **8.1.3**).

8.1.2 *Gloss Stirrer* — of length sufficient for tube *A*, and with its one end bent at right angle in the form of a ring so as to permit a thermometer to pass through the ring.

8.1.3 *Boiling Tube B* — 15 × 4 cm.

8.1.4 *Thermometer* — A long-stem and short-bulb thermometer with a range of 0 to 200°C and with subdivisions at one-tenth of a degree.



All dimensions in millimetres.

FIG. 4 APPARATUS FOR THE DETERMINATION OF SETTING POINT

8.2 Procedure

8.2.1 Place in the boiling tube *A*, a sufficient amount of the sample to give, when melted, a depth of liquid of approximately 7·5 cm. Melt carefully by immersing the tube to a depth of 10 cm in an oil-bath maintained at 14 to 15°C above the anticipated minimum setting point of the test material.

8.2.2 Insert this boiling tube, by means of a cork collar, to within 1·3 cm of the bottom of the boiling tube *B* immersed to a depth of 10 cm in an oil-bath maintained at about 10°C below the anticipated setting point. Place the stirrer in the inner boiling tube. The thermometer is then clamped in a central position with its bulb 2·5 cm from the bottom of the

tube. The temperature of the melt should be approximately 10°C higher than the anticipated setting point at this stage.

8.2.3 Stir at the rate of about two strokes per second by moving the stirrer up and down until the material begins to thicken, at this point stir vigorously, working into the melt portions of the material solidified on the walls of the tube. Stop stirring when the temperature ceases dropping and remains constant for some time (*see* Note). Record this temperature as the setting point.

NOTE — In some instances, there may be a slight rise in the temperature of the material; in such a case, record the highest steady temperature as the setting point.

8.2.3.1 Apply corrections as necessary for the thermometer calibration and the emergent stem.

8.2.3.2 *Emergent stem corrections* — The correction for the emergent stem in mercury-filled thermometers, to be added to the temperature reading, is calculated from the following formula:

$$0{\cdot}000\,15\,N\,(T - t)$$

where

N = number of degrees on the scale of the thermometer between the top of the inner boiling tube and the level of the mercury,

T = temperature reading on the thermometer in the melt, and

t = temperature of the stem at the mid-point of the exposed mercury thread.

9. DETERMINATION OF MATERIAL INSOLUBLE IN ACETONE

9.1 Procedure — Weigh 10 g of the material into a clean and dry 250-ml Erlenmeyer flask fitted with a ground-glass joint. Add 150 ml of dry acetone and warm under reflux until all soluble material is dissolved. Filter the solution using a tared Gooch or sintered-glass crucible of porosity No. 3, and wash well with more solvent. Dry at 110°C for 30 minutes, cool, and then weigh.

9.1.1 For phenyl mercury acetate technical and other similar products containing up to 5 percent matter insoluble in acetone, weigh 2 g of material and proceed in accordance with **9.1**.

9.2 Calculation

$$\text{Matter insoluble in acetone, percent by mass} = \frac{100\,m}{M}$$

where

m = mass in g of the residue obtained after drying, and

M = mass in g of the material taken for the test.

SECTION 2 GENERAL METHODS OF TEST FOR PESTICIDAL FORMULATIONS

10. GENERAL

10.1 The pesticidal formulations, which are commonly used, are water dispersible powders, dusting powders, emulsifiable concentrates and granules.

11. METHODS OF TEST FOR WATER DISPERSIBLE POWDERS

11.1 Sieving Test After Accelerated Storage/Without Pretreatment (*see* Appendix A)

11.1.1 *Apparatus*

11.1.1.1 *Beaker* — of 6·0 to 6·5 cm and 250 ml capacity.

11.1.1.2 *Pressure assembly* — a piston or disc, loosely fitting in the beaker and so formed or weighed as to exert an even pressure of 25 g/cm^2.

11.1.1.3 *Test sieve* — specified IS Sieve [*see* IS : 460 (Part I)-1978*] prepared for the test by removing any film, grease or other water-repellent material and then by drying.

11.1.1.4 *Rubber hose* — of about 10 mm internal diameter.

11.1.2 *Procedure*

11.1.2.1 *Accelerated storage* — Weigh about 20·0 ± 0·5 g of the material in the beaker. Gently level the surface of the material in the beaker with the help of a paper strip, a camel hair brush or a feather. Place the pressure assembly over the surface of the material in the beaker and place it in an oven maintained at 54 ± 1°C for 24 hours. Take out the beaker from the oven, remove the pressure assembly and allow the material in the beaker to come down to room temperature in a closed

*Specification for test sieves : Part I Wire cloth test sieves (*second revision*).

container taking care to avoid unnecessary exposure of the sample to atmospheric humidity.

Formulations of water dispersible powders based on organophosphorus compounds should be placed in 100 ml wide-mouthed neutral glass bottle fitted with a vinyl plastic cap and placed in an oven at temperature and duration as mentioned in Appendix A. Cool the bottle in a closed container before removing the cap. After accelerated storage treatment, the sample should not be exposed to heat, bright sunshine or high atmospheric humidity.

11.1.2.2 *Sieving* — At the completion of 3 hours of cooling weigh accurately about 10·0 g of the treated material (*see* **11.1.2.1**) into a wide-mouth bottle (provided with a cork or rubber stopper) and add 100 ml of tap water. Allow to stand for 30 seconds, then stir for 30 seconds with a 4 to 6 mm diameter glass rod, at not more than 4 rev/s, making no deliberate attempt to break any lumps. Transfer the slurry immediately to the sieve rinsing with tap water. Wash the material on the sieve with an oscillating moderately vigorous spray of tap water issuing out of the rubber hose at the rate of 4 to 5 l/min. Continue washing for 10 minutes or until no more solid particles of the material pass through the sieve. Transfer the residue retained on the sieve to a tared Gooch crucible, dry to constant mass in an oven at a temperature not exceeding 55°C, cool and then weigh. Find the mass of the dry residue.

11.1.3 *Calculation*

$$\text{Material passing through specified IS Sieve, percent by mass} = 100\left(1 - \frac{m}{M}\right)$$

where

m = mass in g of the dry residue obtained (*see* **11.1.2.2**), and

M = mass in g of the material taken for the test.

11.2 Suspensibility Test After Accelerated Storage/Without Pretreatment (*see* Appendix A)

11.2.1 *Apparatus*

11.2.1.1 *Beaker* — *See* **11.1.1.1.**

11.2.1.2 *Pressure assembly* — *See* **11.1.1.2.**

11.2.1.3 *Graduated cylinder* — of capacity 250 ml with a ground glass stopper. The distance between 0- and 250-ml marks shall be 21·0 ± 0·5 cm with a clearance of 6·0 to 8·0 cm between 250-ml mark and the neck of the cylinder (*see* IS : 878-1975*).

*Specification for graduated measuring cylinders (*first revision*).

11.2.1.4 *Glass tube* — About 40 cm long, of an internal diameter of about 5 mm, drawn at one end to an opening of 2 to 3 mm diameter, with the other end connected to a vacuum pump

11.2.2 *Reagent*

11.2.2.1 *Standard hard water* — Dissolve 0·304 0 g of calcium chloride anhydrous and 0·139 0 g of magnesium chlori le hexahydrate in distilled water and make up to one litre in a volumetric flash.

NOTE — Standard hard water is defined as 'water', which provides a hardness of 342 ppm, calculated as calcium carbonate.

11.2.3 *Procedure*

11.2.3.1 *Accelerated storage* — Carry out accelerated storage test as prescribed in **11.1.2.1**.

11.2.3.2 *Suspensibility test* — Weigh accurately into a 100-ml beaker, quantity of the treated/untreated material (*see* **11.2.3.1** and Appendix A) which could form 250 ml of suspension. Add a volume of the standard hard water at 30 ± 1°C equal to at least twice the mass of the material taken for test. Allow to stand for 30 seconds and then stir by hand for 30 seconds with a glass rod 4-6 mm in diameter at not more than, 4 rev/s making no deliberate attempt to break up any lumps. Transfer the slurry to the graduated cylinder. Wash any residue in the beaker with small quantities of standard hard water at 30 ± 1°C. Transfer the washings to the cylinder. Fill the cylinder to the 250-ml mark with standard hard water at 30 ± 1°C. Close the cylinder with the stopper and invert it sharply through 30 complete cycles within one minute, thermally insulating the cylinder from the hands to maintain the temperature of the contents at 30 ± 1°C during the operation. Allow the cylinder to stand at rest for 30 minutes in a waterbath maintained at 30 ± 1°C. (The bench on which the cylinder is placed should be protected from vibrations.) At the end of the settling period (30 minutes), dip the nozzle of the glass tube into the supernatant liquid contained in the cylinder and withdraw nine-tenths (225 ml) of the suspension within 10 to 15 seconds, using the vacuum pump. During its withdrawal, the suspension should be disturbed as little as possible, which could be achieved by maintaining the nozzle of the glass tube just below the sinking top level of the suspension. Discard the suspension so withdrawn. The sediment at the bottom of the cylinder shall not exceed 25 ml.

NOTE — Should excessive flocculation occur during the test, the material shall be deemed unsatisfactory. Excessive flocculation in this context means either curdling up of the suspension in the entire column or the appearance of the transparent liquid accompanied by total or partial curdling up.

11.2.3.3 Proceed with the retained one-tenth of the suspension, including the sediment for the determination of active ingredient in accordance with the guidelines given in Appendix A.

11.2.4 *Calculation*

$$\text{Suspensibility, percent by mass} = \frac{1\,000\,(M - m)}{9M}$$

where

M = mass in g of pesticide present in the material taken for the preparation of the suspension (*see* **11.2.3.2**), and

m = mass in g of pesticide found in the suspension including the sediment remaining in the graduated cylinder (*see* **11.2.3.3**).

11.3 Acidity or Alkalinity Test

11.3.1 *Qualitative Test*

11.3.1.1 *Procedure* — Take about 0·5 g of the material in a test-tube and mix with about 1 ml of water. Test the mixture for acidity or alkalinity with a litmus paper. Determine the acidity (*see* **11.3.2**) or the alkalinity (*see* **11.3.3**), as the case may be.

11.3.2 *Determination of Acidity*

11.3.2.1 *Reagents*

a) *Methyl red indicator solution-aqueous* — one percent (*m/v*).

b) *Bromocresol purple indicator solution* — one percent (*m/v*) in ethyl alcohol.

c) *Standard sodium hydroxide solution* — 0·05 N.

d) *Standard hydrochloric acid* — 0·05 N.

11.3.2.2 *Procedure* — Weigh accurately 10·0 g of the material into a dry conical flask, add 25 ml of acetone and mix. Warm the flask gently to effect the solution of the active ingredient present. Add 75 ml of water and let it stand for an hour. Filter the supernatant aqueous extract and take 50 ml filtrate. Titrate immediately with the standard sodium hydroxide solution using methyl red or bromocresol purple as the indicator. Alternatively, the end point may be determined electrometrically (*see* **13.5.4**).

Carry out a blank determination on an aliquot of 50 ml made from 25 ml acetone and 75 ml water.

11.3.2.3 *Calculation*

$$\text{Acidity (as } H_2SO_4\text{), percent by mass} = \frac{4{\cdot}9 \times 2(V - v)N}{M}$$

where

V = volume in ml of the standard sodium hydroxide solution required for the test with the material,

v = volume in ml of the standard sodium hydroxide solution required for the blank determination,

N = normality of the standard sodium hydroxide solution, and

M = mass in g of the material taken for the test.

In case the blank shows alkaline reaction, neutralize with the standard hydrochloric acid and calculate the acidity as follows:

$$\text{Acidity (as } H_2SO_4\text{), percent by mass} = \frac{4{\cdot}9 \times 2(VN_1 + vN_2)}{M}$$

where

V = volume in ml of the standard sodium hydroxide solution required for the test with the material,

N_1 = normality of the standard sodium hydroxide solution,

v = volume in ml of the standard hydrochloric acid required for the blank determination,

N_2 = normality of the standard hydrochloric acid, and

M = mass in g of the material taken for the test.

11.3.3 *Determination of Alkalinity*

11.3.3.1 *Reagents*

a) *Methyl red indicator solution* — aqueous, one percent (*m/v*).

b) *Bromocresol purple indicator solution* — one percent (*m/v*) in ethyl alcohol.

c) *Standard hydrochloric acid* — 0·05 N.

d) *Standard sodium hydroxide solution* — 0·05 N.

11.3.3.2 *Procedure* — Weigh accurately 10·0 g of the material into a dry conical flask, add 25 ml of acetone and mix. Warm the flask gently to effect the solution of the active ingredient present. Add 75 ml of

water and let it stand for an hour. Filter the supernatant aqueous extract and take 50 ml of filterate. Titrate immediately with the standard hydrochloric acid using methyl red or bromocresol indicator as the indicator. Alternatively, the end point may be determined electrometrically (*see* **13.5.4**)

Carry out a blank determination on 50 ml aliquot made from 25 ml acetone and 75 ml water.

11.3.3.3 *Calculation*

$$\text{Alkalinity (as NaOH), percent by mass} = \frac{4{\cdot}0 \times 2\,(V - v)\,N}{M}$$

where

V = volume in ml of the standard hydrochloric acid required for the test with the material,

v = volume in ml of the standard hydrochloric acid required for the blank determination,

N = normality of the standard hydrochloric acid, and

M = mass in g of the material taken for the test.

In case the blank shows acid reaction, neutralize with the standard sodium hydroxide solution and calculate the alkalinity as follows:

$$\text{Alkalinity (as NaOH), percent by mass} = \frac{4{\cdot}0 \times 2\,(VN_1 + vN_2)}{M}$$

where

V = volume in ml of the standard hydrochloric acid required for the test with the material,

N_1 = normality of the standard hydrochloric acid,

v = volume in ml of the standard sodium hydroxide solution required for the blank determination,

N_2 = normality of the standard sodium hydroxide solution, and

M = mass in g of the material taken for the test.

11.4 Wettability Test

11.4.1 *Apparatus*

11.4.1.1 *Beaker* — *See* **11.1.1.1.**

11.4.2 *Procedure* — Weigh accurately about 2 g of the material and pour uniformly and quickly in the beaker containing 100 ml of the standard hard water (*see* **11.2.2.1**). Start the stop-watch simultaneously. Note the time when the whole material is completely submerged into the water.

12. METHODS OF TEST FOR DUSTING POWDERS

12.1 Test for Sieving Requirement for Particle Size

12.1.1 *Apparatus*

12.1.1.1 *Test sieve* — *See* **11.1.1.3**.

12.1.2 *Procedure*

12.1.2.1 *Dusting powders* — Weigh accurately 10·0 g of the material and transfer it to the test sieve. Cover the sieve and screen the material in a Ro-Tap or a similar machine for 20 minutes. Two small square rubber cubes are introduced along with the material on the sieve to facilitate the breaking up of any soft lumps of the caked material. After 20 minutes stop the machine and brush the residue on the sieve into a tared weighing dish. Weigh the dish and determine the mass of the residue.

12.1.2.2 *Granules* — Weigh 100 g of the material and transfer to a deck of sieves consisting of sieves of upper declared limit, lower declared limit and a receiver. Screen for 15 minutes. Collect separately the fractions retained on sieves. Weigh separately fractions retained on the sieve of upper declared limit and collected in the receiver. Determine the oversize retained on the sieve of upper declared limit and that passing through the sieve of lower declared limit.

12.1.3 *Calculation*

Material passing through specified

$$\text{IS Sieve, percent by mass} = 100\left(1 - \frac{m}{M}\right)$$

where

m = mass in g of the material retained on the test sieve, and

M = mass in g of the material taken for the test.

12.2 Test for Bulk Density After Compacting

12.2.1 *Apparatus*

12.2.1.1 *Graduated cylinder* — of 100 ml capacity (*see* IS : 878-1975*) with internal diameter of 27 to 29 mm.

*Specification for graduated measuring cylinders (*first revision*).

12.2.1.2 *Funnel* — wide and short-stemmed.

12.2.2 *Procedure*

12.2.2.1 *Bulk density before compacting* — Rest the funnel over the top edge of the tared graduated cylinder. Fill the cylinder to the 100-ml mark by pouring the material through the funnel, without tapping, and level the powder with the minimum of disturbance. Leave the cylinder untouched for 5 minutes and add more powder if necessary, to bring the contents to the 100-ml mark, level again with the minimum of disturbance. Weigh the filled cylinder and calculate the bulk density before compacting (mass of the material/volume occupied by the material).

12.2.2.2 *Bulk density after compacting* — Stopper the filled cylinder (*see* **12.2.2.1**), and drop it 20 times through a height of 15 cm on to a felt pad resting on a hard surface. Note the volume of the material after compacting. Calculate the bulk density of the material after compacting (mass of the material/volume occupied by the material after compacting).

12.2.3 *Test Evaluation* — The value obtained in **12.2.2.2** shall not exceed the value obtained in **12.2.2.1** by more than 60 percent.

12.3 Test for Acidity or Alkalinity — The test for acidity or alkalinity determination shall be conducted on the same lines as given under **11.3.**

13. METHODS OF TEST FOR EMULSIFIABLE CONCENTRATES WATER SOLUBLE CONCENTRATES

13.1 Cold Test

13.1.1 *Procedure* — Take 50 ml of the material in a 100-ml transparent glass container and close it with a cork or stopper fitted with the thermometer. Cool the material to 10°C (*see* Note) by placing the container in ice cold water. When the temperature of the material in the container reaches 10°C, add to it a small seeding crystal of the pesticide with minimum opening of the stopper within the shortest possible duration of time. In case the formulation has been prepared from technical liquid pesticides, there is no need to seed with crystal. Gently stir the material in the container at short intervals for one hour maintaining the temperature of the material at 10°C. At the end of one hour, examine the material for any turbidity or separated solid or oily matter or both.

NOTE — In case the purchaser specifies that the test is required to be carried out at 0°C, the material in a container shall be cooled to 0°C by placing the container in a bath of crushed ice and the test carried out at 0°C instead of 10°C.

13.2 Flash Point (Abel) — When determined by the method as prescribed in IS : 1448 [P : 20]-1960*, the flash point of the material shall be above 24·5°C.

13.3 Test for Emulsion Stability

13.3.1 *Methods* — Two methods are prescribed for the determination of emulsion stability of the material. The method to be employed shall depend upon the manufacturer's instructions for the preparation of the diluted emulsion from the material. Method 1 shall be employed for material which is required to be added to standard hard water for the preparation of the diluted emulsion, while Method 2 shall be employed for material to which standard hard water should be added to prepare the diluted emulsion.

13.3.2 *Method 1*

13.3.2.1 *Apparatus*

a) *Beaker* — capacity 250 ml, with an internal diameter of 6·0 to 6·5 cm and marked at 100 ml,

b) *Mohr-type pipette*,

c) *Glass-rod* — 4 to 6 mm in diameter and of a convenient length, and

d) *Graduated cylinder* — capacity 100 ml.

13.3.2.2 *Reagent*

a) *Standard hard water* — *See* **11.2.2.1**.

13.3.2.3 *Procedure* — Pour 75 to 80 ml of standard hard water, brought to a temperature of 30 ± 1°C, in the beaker. By means of the pipette add 5 ml of the concentrate if meant for public health use and 2 ml of the concentrate if meant for agricultural use, while stirring with a glass rod, at about 4 rev/s. The concentrate should be added to water at the rate of 25 to 30 ml/min, with the point of the pipette 2 cm inside the beaker, the flow of the concentrate being directed towards the centre, and not against the sides of the beaker. Make up to 100 ml with water with continuous stirring and immediately pour into the clean and dry cylinder. The stirring time should be 3 minutes from the beginning of the addition of the concentrate until the emulsion is poured into the 100-ml cylinder. Keep at 30 ± 1°C for one hour and examine for any creaming or separation.

*Methods of test for petroleum and its products: P : 20 Flash point by Abel apparatus.

13.3.2.4 *Report* — Report the total volume in ml of the creamed matter at the top and the sediment at the bottom in the cylinder.

13.3.3 *Method 2*

13.3.3.1 *Apparatus*

a) *Beaker — See* **11.1.1.1**,

b) *Dropping funnel*,

c) *Glass rod — See* **13.3.2.1** (c), and

d) *Graduated cylinder — See* **13.3.2.1** (d).

13.3.3.2 *Reagent*

a) *Standard hard water — See* **11.2.2.1**.

13.3.3.3 *Procedure* — Take 5 ml of concentrate if meant for public health use and 2 ml of concentrate if meant for agricultural use. Pour standard hard water at 30 ± 1°C into the droping funnel, and add it to the material contained in the beaker at the rate of 15 to 20 ml/min. During the addition, stir the contents of the beaker continuously with the glass rod, and stop the addition of standard hard water just when the volume of diluted emulsion in the beaker reaches 100 ml. Transfer the diluted emulsion immediately to the clean and dry graduated cylinder. Keep the cylinder with its contents at 30 ± 1°C for one hour. After this period, note the volume of the creamed matter at the top and the sediment at the bottom, if any.

13.3.3.4 *Report* — Report the total volume in ml of the creamed matter at the top and the sediment at the bottom in the cylinder.

13.4 Test for Heat Stability

13.4.1 *Apparatus*

13.4.1.1 *Glass bottle* — of convenient size, with an air-tight glass stopper.

13.4.1.2 *Thermostat* — capable of maintaining the temperature at 54 ± 1°C.

13.4.2 *Procedure*

13.4.2.1 Pour the material into the glass bottle until it is about three-fourths full. Stopper the bottle and seal it air-tight with sealing wax to avoid any loss of the volatile solvent. Place the filled bottle in the thermostat maintained at 54 ± 1°C for 24 hours. Remove the bottle from the thermostat after 24 hours and cool its contents to room temperature.

13.4.2.2 Test the treated material for compliance with the requirements of cold test, flash-point test and emulsion-stability test.

NOTE — This test is done on emulsifiable concentrates of certain pesticides only.

13.5 Acidity or Alkalinity Test

13.5.1 *Qualitative Test*

13.5.1.1 *Procedure* — Take about 0·5 ml of the material in a test tube and mix with about one millilitre of water. Test the mixture for acidity or alkalinity with a litmus paper. Determine, as the case may be, the acidity (*see* **13.5.2**) or the alkalinity (*see* **13.5.3**).

13.5.2 *Determination of Acidity*

13.5.2.1 *Reagents*

a) *Methyl red indicator solution* — aqueous one percent (*m/v*).

b) *Bromocresol purple indicator solution* — one percent (*m/v*) in ethyl alcohol.

c) *Standard sodium hydroxide solution* — 0·05 N.

d) *Standard hydrochloric acid* — 0·05 N.

13.5.2.2 *Procedure* — Weigh accurately about 10 g of the material into a dry conical flask and dilute with 100 ml of water. Titrate the contents of the flask immediately with the standard sodium hydroxide solution using methyl red or bromocresol purple as the indicator. Alternatively, the end-point may be determined potentiometrically (*see* **13.5.4**).

Carry out a blank determination on 100 ml of water.

13.5.2.3 *Calculation*

$$\text{Acidity (as } H_2SO_4\text{), percent by mass} = \frac{4{\cdot}9\,(V - v)\,\mathcal{N}}{M}$$

where

V = volume in ml of the standard sodium hydroxide solution required for the test,

v = volume in ml of the standard sodium hydroxide solution required for the blank determination,

$\mathcal{N}$ = normality of the standard sodium hydroxide solution, and

M = mass in g of the material taken for the test.

In case the blank determination shows alkaline reaction, neutralize with the standard hydrochloric acid and calculate the acidity as follows:

$$\text{Acidity (as } H_2SO_4\text{), percent by mass} = \frac{4{\cdot}9\,(V N_1 + v N_2)}{M}$$

where

V = volume in ml of the standard sodium hydroxide solution required for the test,

N_1 = normality of the standard sodium hydroxide solution,

v = volume in ml of the standard hydrochloric acid required for the blank determination,

N_2 = normality of the standard hydrochloric acid, and

M = mass in g of the material taken for the test.

13.5.3 *Determination of Alkalinity*

13.5.3.1 *Reagents*

a) *Methyl red indicator solution* — Aqueous one percent (*m/v*).

b) *Bromocresol purple indicator solution* — One percent (*m/v*) in ethyl alcohol.

c) *Standard hydrochloric acid* — 0·05 N.

d) *Standard sodium hydroxide solution* — 0·05 N.

13.5.3.2 *Procedure* — Weigh accurately about 10 g of the material into a dry conical flask and dilute with 100 ml of water. Titrate the contents of the flask immediately with the standard hydrochloric acid, using methyl red or bromocresol as the indicator. Alternatively, the end-point may be determined potentiometrically.

Carry out a blank determination with 100 ml of water.

13.5.3.3 *Calculation*

$$\text{Alkalinity (as NaOH), percent by mass} = \frac{4{\cdot}0\,(V - v)\,N}{M}$$

where

V = volume in ml of the standard hydrochloric acid required for the test,

v = volume in ml of the standard hydrochloric acid required for the blank determination,

N = normality of the standard hydrochloric acid, and

M = mass in g of the material taken for the test.

In case the blank determination shows an acid reaction, neutralize with the standard sodium hydroxide solution and calculate the alkalinity as follows:

$$\text{Alkalinity (as NaOH), percent by mass} = \frac{4{\cdot}0\,(VN_1 + vN_2)}{M}$$

where

V = volume in ml of the standard hydrochloric acid for the test,

N_1 = normality of the standard hydrochloric acid,

v = volume in ml of the standard sodium hydroxide solution required for the blank determination,

N_2 = normality of the standard sodium hydroxide solution, and

M = mass in g of the material taken for the test.

13.5.4 *Electrometric Procedure for Determination of Acidity or Alkalinity*

13.5.4.1 *Reagents*

a) *Buffer-solution* — Mix acetic acid (100 ml of 2 N solution) and sodium hydroxide (100 ml of 1N solution) and make up to one litre with demineralized water.

b) *Acetone* — distilled.

c) *Sodium hydroxide* — 0·05 N.

d) *Hydrochloric acid* — 0·05 N.

13.5.4.2 *Apparatus*

a) *p*H meter.

13.5.4.3 *Procedure* — Determine the apparent *p*H, at 20°C, of a mixture of the acetone (50 ml) and buffer solution (5 ml), with the electrodes and *p*H meter to be used in the titration. Transfer 10 g of the sample to the conical flask. Add acetone (75 ml) and stir for 5 minutes. Filter through a Buchner funnel into the filter flask. Rinse the conical flask and the Buchner funnel with acetone (4 × 5 ml). Transfer the combined acetone extracts to the titration vessel rinsing the filter flask with acetone (5 ml). Add distilled water (10 ml) and titrate electrometrically with sodium hydroxide solution or hydrochloric acid to

the apparent *p*H of acetone/buffer mixture at 20°C. Calculate the acidity or alkalinity whatsoever by the factor given in **13.5.2.3** or **13.5.3.3.**

14. METHODS OF TEST FOR GRANULES

14.1 Encapsulation

14.1.1 *Attrition Test*

14.1.1.1 *Procedure* — Take 100 g of the material and sieve it for 6 hours in 75-micron IS Sieve [*see* IS : 460 (Part I)-1978*] in a ro-tap machine.

14.1.1.2 Collect the residue from the test sieve and determine its active ingredient by the method prescribed in the relevant Indian Standard.

14.1.2 *Water Run-Off Test*

14.1.2.1 *Procedure* — Take 10 g of the material in a 100 ml capacity burette plugged with cotton/glass wool at the bottom and add 50 ml water. Keep it for 15 minutes. Drain off the water after 15 minutes.

14.1.2.2 Determine active ingredient in the drained water so collected (*see* **14.1.2.1**) by the method prescribed in the relevant Indian Standard.

14.1.3 *Wet Test for Encapsulation*

14.1.3.1 *Reagents*

a) Solvent same as used for the determination of active ingredient,

b) *Sodium sulphate* — anhydrous.

14.1.3.2 *Apparatus*

a) *Wide mouth bottle* — 15 cm in length and 11 cm in diameter.

b) *Roller pot mill*

c) *Cloth piece* — 30 × 15 cm. Grey medium-B.

14.1.3.3 *Procedure* — Moisten the cloth piece with 6 ml water and place it in the glass bottle. Place 100 g of the material in it and close the bottle. Rotate the bottle on a roller mill at 50 RPM for two and a half hours. Take out the cloth and remove all granules adhering to the cloth by jerking the cloth. Place the cloth piece in a 250-ml beaker. Add 100 ml methylene chloride or any other specified solvent into the beaker and let it stand for 30 minutes, then swirl the cloth piece with the

*Specification for test sieves: Part I Wire cloth test sieves (*second revision*).

help of a glass rod and decant the extract through a bed of anhydrous sodium sulphate contained in a funnel fitted with a cotton plug, into a clean 250-ml round bottom flask. Wash the cloth twice with 50-ml portions of methylene chloride each time and decant the washings through the same sodium sulphate bed into the same flask. Add one drop of diethylene glycol and attach the flask to a rotary vacuum evaporator with vacuum pre-set at 50 mm of Hg. Immerse the flask in a 45°C waterbath and strip the solvent to complete dryness. Add 2 ml of specified solvent to the flask and swirl to effect complete dissolution. Transfer the contents of the flask to a 10-ml volumetric flask and rinse the flask repeatedly with small portions of specified solvent. Transfer washings to the same 10-ml volumetric flask and make up the volume. Determine the active content in this solution by the method prescribed in the relevant Indian Standard.

14.1.3.4 *Calculation*

$$\text{Active content released, percent by mass} = \frac{M_1 \times 10}{M_2}$$

where

M_1 = concentration of active content in solution, and

M_2 = active content in granules.

14.1.4 *Liquid Holding Capacity (LHC)*

14.1.4.1 *Principle* — This characteristic determines the maximum loading of a technical formulation that can be put on a given type of blank granule.

14.1.4.2 *Procedure* — Make up a mixture of heavy aromatic naphtha and cyclohexanone to a relative density of 1·000 (alternatively a mixture of monochlorobenzene and xylene can be used). Fill this mixture of organic solvents in a burette.

Take 20 g of sample in a 250-ml bottle. Add solvent mixture from burette drop by drop, into the centre of the sample, shake the bottle. Continue to add solvent, shaking the bottle till some particles of the sample begin to stick on the sides of the bottle.

14.1.4.3 *Calculation*

$$\text{Liquid holding capacity, percent by mass} = \frac{M}{M_1 + M} \times 100$$

where

M = mass of solvent, in g, used; and

M_1 = mass of blank granules taken for the test.

APPENDIX A

(*Clauses* 11.1 *and* 11.2)

ACCELERATED STORAGE, CONCENTRATIONS OF ACTIVE INGREDIENTS REQUIRED FOR SUSPENSIBILITY AND TREATMENT OF SUSPENSION INCLUDING SEDIMENT

Sl. No.	Formulation	Concentration	Suspensibility/ Sieving Test	Treatment of Suspension and Sediment
(1)	(2)	(3)	(4)	(5)
i)	BHC WDP	0·5 percent gamma *m/v*	After accelerated storage at 54 ± 1°C for 24 hours	Shake vigorously the remaining one tenth suspension including sediment with sufficient acetone. Make the volume to 250 ml. Take a suitable aliquot and determine the active ingredient by the hydrolysable chlorine method prescribed in Appendix A of IS : 561-1978.
ii)	Carbaryl WDP	0·5 percent *m/v*	After accelerated storage at 54 ± 1°C for 24 hours	Shake vigorously the remaining one tenth suspension including sediment with sufficient methanol and make up the volume to 250 ml in the suspensibility cylinder. Take a suitable aliquot for the determination of active content by the method prescribed in Appendix A of IS : 7121-1973.
iii)	Carbendazim WDP	0·5 percent *m/v*	After accelerated storage at 54 ± 1°C for 24 hours	Filter the sediment including suspension using distilled water. Dissolve the sediment in a mixture of acetic acid and acetic anhydride (90 : 10), making the volume

				to 100 ml using same mixture. Take suitable aliquot in a ground-glass flask and stopper it. Reflux on a water bath for 10 minutes and then proceed by the method as prescribed in Appendix A of IS : 8446-1975.
iv)	Copper oxychloride WDP	0·5 percent *m/v*	Without pretreatment	Transfer quantitatively the remaining suspension and sediment to an Erlenmeyer flask and determine the active ingredient by the method prescribed in Appendix A of IS : 1506-1977.
v)	Cuprous-oxide WDP	0·5 percent *m/v*	Without pretreatment	
vi)	DDT WDP	2·5 percent *m/v*	After accelerated storage at 54 ± 1°C	Shake vigorously the remaining one tenth suspension including sediment with sufficient acetone and make the volume to 250 ml. Take suitable aliquot and determine the active ingredient content by the method prescribed in Appendix A of IS : 565-1975.
vii)	Diazinon WDP	0·5 percent *m/v*	After accelerated storage at 54 ± 1°C for 24 hours	Transfer the retained one tenth of suspension including sediment, to a 250 ml separating funnel. Rinse the cylinder twice with 50 ml portions of petroleum ether (b.p. 40-60°C), adding the rinsings to separating funnel. Shake and drain the aqueous layer into a second 250 ml separating funnel. Add to the second separating funnel 100 ml of light petroleum and 10 ml of saturated sodium chloride solution and shake again. Let the phases separate and then discard the aqueous phase. Combine the petroleum ether extracts and then proceed as per Appendix A of IS : 2862-1964.

viii)	Endosulfan WDP	0·25 percent *m/v*	After accelerated storage at 54 ± 1°C	Transfer the suspension including sediment to the reaction flask. Wash the cylinder thrice with methanol using a quantity of not more than 100 ml, and then determine the active ingredient content by the method prescribed in Appendix A of IS : 4324-1967.
ix)	Malathion WDP	2·5 percent *m/v*	After accelerated storage at 90 ± 2°C	Instead of 250 ml suspension 100 ml suspension is prepared. After 30 minutes 25 ml aliquot is withdrawn from the level of 50 ml. Malathion content is determined in this aliquot. Details of the procedure are given in Amendment No. 2 issued to IS : 2569-1978.
x)	Mancozeb WDP	0·5 percent *m/v*	After accelerated storage at 54 ± 1°C for 24 hours	Transfer quantitatively the retained one tenth portion of suspension including sediment to a reaction flask, wash the cylinder with minimum quantity of water and add washings to the reaction flask. Proceed for the determination of active ingredient content by the method prescribed in Appendix A of IS : 8707-1978.
xi)	Sulphur wettable powder	0·5 percent *m/v*	After accelerated storage at 54 ± 1°C for 24 hours	Transfer the remaining one tenth suspension including sediment, quantitatively, using the minimum amount of water, to a 300-ml conical flask and determine the active ingredient content by the method prescribed in Appendix A of IS : 3383-1975.

xii)	Thiram WDP	0·5 percent *m/v*	After accelerated storage at 54 ± 1°C	Transfer the remaining one tenth suspension including sediment, quantitatively, using minimum amount of water to a reaction flask and then determine the active ingredient by the method prescribed in Appendix A of IS : 4766-1968.
xiii)	Zineb WDP	0·5 percent *m/v*	After accelerated storage at 54 ± 1°C for 24 hours	Transfer the retained one tenth suspension, including sediment, quantitatively, using minimum quantity of water to a reaction flask and then proceed by the method prescribed in Appendix A of IS : 3898-1981.
xiv)	Ziram WDP	2·5 percent *m/v*	After accelerated storage at 34 ± 1°C for 54 hours	Transfer the retained one tenth suspension including sediment, quantitatively, using minimum quantity of water to a reaction flask and determine the active ingredient content by the method prescribed in Appendix A of IS : 3900-1975.

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